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Hot Filament Deposition of Diamond Films

by

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Effects of multiple filament geometry in the hot filament deposition of diamond films

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Abstract

Scaleup of hot filament chemical vapor deposition of diamond films requires knowledge of how filament geometry affects the deposition process. The effect of multiple filament geometry on the growth rate and surface morphology of diamond films is presented. Three factors were varied: the number of helical turns in a filament - 5, 10 or 15; the number of filaments present -- 1 or 2; and the CH_4 fraction in the CH_4 - H_2 feed gas mixture -- 0.25%, 0.5%, 0.75% or 1.0%. Other deposition parameters were 750 °C substrate temperature, 52 standard cm^3/m total feed gas flow rate, and 5300 Pa deposition pressure. The deposition chamber volume was 1.9 liters. Increasing the CH_4 concentration in the feed gas results in higher growth rates. However, increasing the number of filament turns in dual filaments systems resulted in lower than expected growth rates for dual 10 and 15 turn filaments. Those films that grew at lower than expected growth rates also display morphologies typical of films produced at lower CH_4 concentrations. These effects may be due to saturation of those species participating in diamond formation with increase in filament surface area and CH_4 gas fraction. The substrate surface area coverage of dual filament depositions was double that of the single filament depositions.

1. INTRODUCTION

In order to increase the deposition area in hot filament chemical deposition (HFCVD) of diamond films, one must understand how the filament geometry will affect the growth rate and morphology of the deposited films. In this paper we report on the results of depositions carried out under different deposition conditions. Three factors were varied: The number of filaments (1 or 2), the number of helical turns in a filament (5, 10 or 15), and the CH_4 fraction in the CH_4 - H_2 feed gas mixture. While the general trend was an increase in the growth rate with increasing methane fraction and increasing number of filament turns, using dual filaments rather than a single filament resulted in lower than expected growth rates for dual 10 and 15 turn filament. Lower growth rates always occurred when the filaments contained a high number of turns (15 turns). Changes in the morphology of the deposited films accompanied changes in the filament geometry. It was observed the surface area coverage of dual filament depositions was double that of the single filament depositions. This paper is an extension of work reported earlier.[1]

2. EXPERIMENTAL

A bell jar hot filament deposition chamber (1.5 liters) described previously [2] was used for the diamond deposition. Filaments were wound into helical shapes from 0.12 mm diameter tungsten wire with ~ 1 mm turn diameter. The length of the helical shape was ~ 2 cm for all filaments. Five, 10 and 15 turn filaments were used. Filaments were cured using typical deposition parameters for 24 h before being used in an actual deposition. The distance between substrate and filament was 4 mm.

Single crystal Si substrates 1 cm x 1 cm were used. Prior to all depositions, the substrates were rubbed with 1 μ m diamond paste and cleaned. Four substrates (arranged in a square pattern with two substrates centered under each filament) were used in each dual filament deposition; two substrates were used for single filament depositions, the substrates being centered under the single filament. Average thicknesses were calculated by weighing the specimens before and after deposition. Average thicknesses for a deposition varied no more than 15%.

The deposition parameters were: nominal filament temperature 1800°C; nominal substrate temperature 750°C; deposition pressure 5300 Pa (40 torr); and, feed gas flow, 52 standard cm³/m. CH₄ fractions in the CH₄-H₂ feed gas were 0.25%, 0.50%, 0.75% and 1.0%. The deposition times were 48 h except for the single 5 turn filament depositions which were 72 h due to the lower deposition rates.

3. RESULTS AND DISCUSSION

The effect of the number of turns in the filament at different methane concentrations on the growth rates for single and dual filament systems are shown in Figure 1. The highest growth rate occurred in a deposition using a 15 turn single filament with a CH₄ fraction of 1.0%. For single filament depositions the growth rate increases approximately linearly with the number of turns. However, for dual filaments, the growth rates are approximately independent of the number of turns. These results show that simply increasing the number of filament turns by introducing dual filaments does not necessarily increase the growth rate. Depletion and saturation effects must also be considered.

In Figures 2-7, the effect of CH₄ fraction on the diamond film growth rate and morphology are shown for single and dual filament systems. Figure 2 shows previous data on the growth rate of single and dual 5 turn filament vs. CH₄ fraction in the feed gas [1]. The growth rate increases nearly linearly with increasing methane concentration for both dual and single filament configurations and the dual filament gives a higher growth rate. Figure 3 shows scanning electron microscope (SEM) micrographs of the surface morphology of the single five turn filament depositions. (Dual filament micrographs were shown previously [1].) The predominant effect observed is an increase in the secondary nucleation with increasing CH₄ fraction. For 1.0% methane deposition (Figure 3d), the secondary nucleation has all but obscured the faceting of the film. In the 0.75% deposition, Figure 3c, mostly (111) and some (100) facets can be observed. The (100) habit has been seen in microwave assisted chemical vapor deposition (MACVD) of diamond using methane rich feed gas mixtures [3-6]. The heavy secondary nucleation contrasts with the fairly well faceted (100) surfaces observed in dual five turn filament depositions [1]. The decrease

in the secondary nucleation in the dual filament system suggests a possible depletion of diamond forming species.

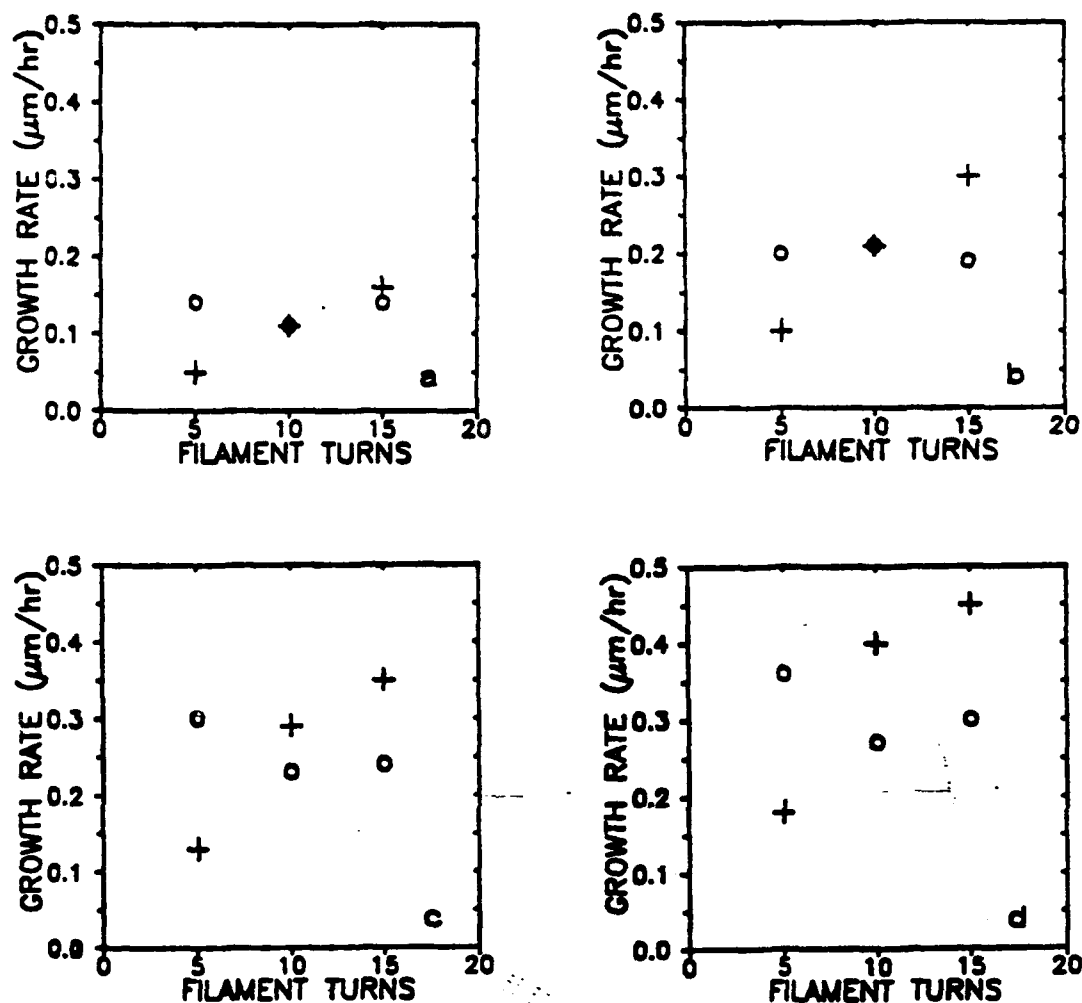


Figure 1. Growth rate vs. number of turns for dual (○) and single (+) filament systems for $\text{CH}_4:\text{H}_2$ of (a) 0.25%, (b) 0.5%, (c) 0.75% and (d) 1.0%.

Figure 4 shows the effect of CH_4 fraction on the growth rates of depositions using single and dual 10 turn filament systems. At the lowest CH_4 fractions (0.25%, 0.50%) growth rates for dual and single filament systems are equal while at the highest CH_4 fractions (0.75%, 1.0%) the dual filament system yielded lower growth rates. In Figure 5 shows SEM micrographs of the surface morphologies of the single 10 turn filament depositions. (Dual filament micrographs were shown previously [1].) Increasing the CH_4 fraction from 0.25% to 1.0% (Figure 5a-d), changes the surface morphology from predominately (111) habit to a predominantly (100) habit. An increase in secondary nucleation is also noted with increasing CH_4 fraction. The surface morphologies of single and dual filament depositions [1] were quite similar.

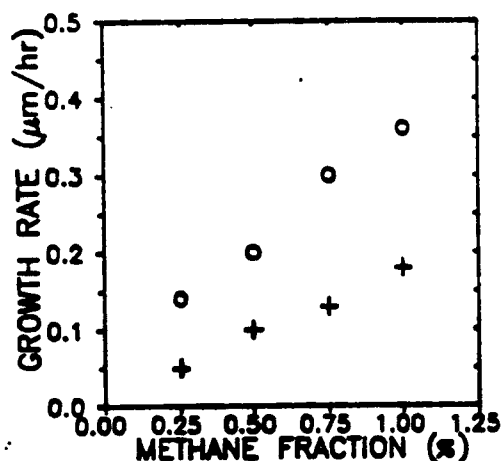


Figure 2. Growth rate vs. % CH₄. For dual (○) and single (+) 5 turn filaments.

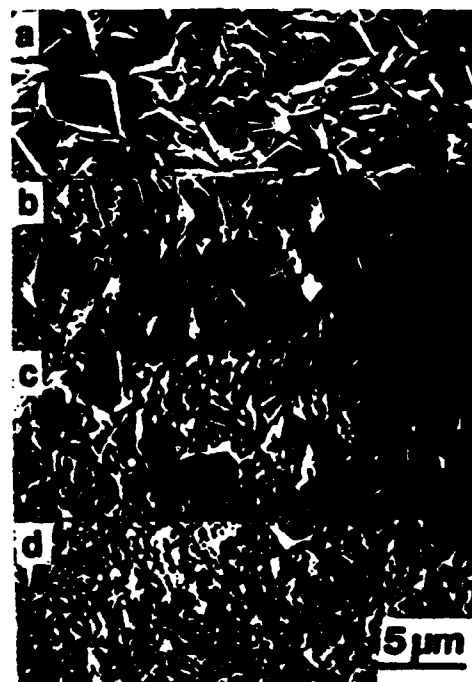


Figure 3. SEM micrographs of films deposited using single 5 turn filaments for CH₄:H₂ of (a) 0.25%, (b) 0.5%, (c) 0.75% and (d) 1.0%.

Figure 6 shows the effect of CH₄ fraction on the growth rates of depositions using single and dual 15 turn filament systems. The dual filament deposition rates were less than the single filament deposition rates at a given feed gas composition. Furthermore, the difference between the single dual filament deposition rates increased with increasing methane concentration. Figure 7 shows SEM micrographs of the surface morphologies of the single 15 turn filament depositions. (Dual filament micrographs were shown previously [1].) There is little change in the morphologies going from 0.25% to 1.0% CH₄ fraction (Figure 7a-d). Mainly (111) and (220) habits are seen in these micrographs and in the micrographs of dual 15 turn depositions [1]. With 1.0% CH₄ fractions in both single and dual 15 turn filament depositions the faceting is typical of faceting observed on films deposited at the lower CH₄ fractions.

Comparing the figures it is seen that only for the five turn filament does the dual filament system produce a growth rate which exceeds that of the single filament deposition.

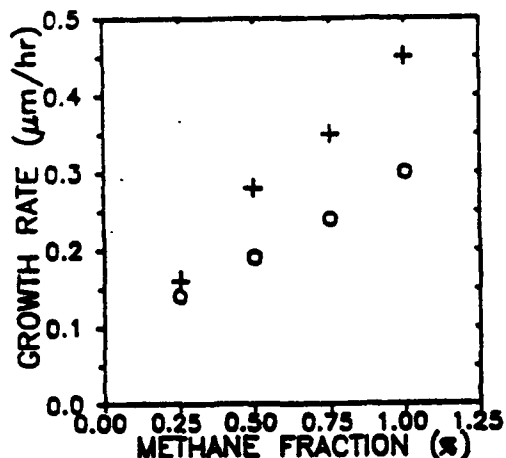


Figure 4. Growth rate vs. % CH_4 for dual (O) and single (+) 10 filaments.



Figure 5. SEM micrographs of films deposited using single 10 turn filaments for $\text{CH}_4:\text{H}_2$ of (a) 0.25%, (b) 0.5%, (c) 0.75% and (d) 1.0%

4. CONCLUSIONS

CVD diamond depositions were carried out in a hot filament reactor with single and dual filaments. In the 5 turn systems, dual filaments produce higher growth rates than single filaments at a specific feed gas composition; in the 10 turn systems, the dual filament systems exhibit growth rates equal to or lower than those in single filament systems at a specific feed gas composition; and, in 15 turn systems, the dual filament systems exhibit lower growth rates than those single filament systems at specific feed gas composition. Surface faceting in films deposited with the highest methane concentrations and the highest number of filament turns are typical of faceting in films deposited with lower methane concentrations. These effects can possibly be related to differences in gas phase reaction kinetics, differences in active filament surface areas, and gas temperature changes in the reaction zone. These factors can cause depletion and saturation of different species participating in diamond formation. For all depositions the surface area of continuous film coverage for dual filament depositions was double that of single filament depositions.

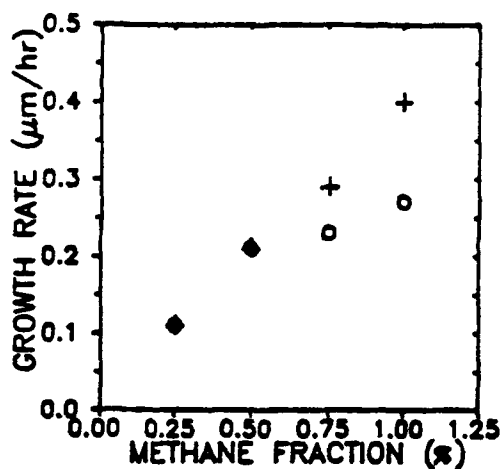


Figure 6. Growth rate vs. % CH₄ for dual (O) and single (+) 15 turn filaments.



Figure 7. SEM Micrographs of films deposited using single 15 turn filaments for CH₄:H₂ of (a) 0.25%, (b) 0.5%, (c) 0.75% and (d) 1.0%.

5. ACKNOWLEDGEMENT

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6. REFERENCES

- 1 E. N. Farabaugh, A. Feldman, and L. Robins, Proc. 2nd Inter. Conf. on the New Diamond Science and Technology (in press).
- 2 E. N. Farabaugh, A. Feldman, Mater. Res. Soc. Proc., 162 (1990) 127.
- 3 B. E. Williams, H. S. Kong, and J. T. Glase, J. Mater. Res., 5 (1990) 801.
- 4 N. Seleka, J. Mater. Res., 4 (1989) 668.
- 5 W. Zu, A. R. Badzian, and R. Messier, SPIE Proc., 1325 (1990) 187.
- 6 B. E. Williams and J. T. Glass, J. Mater. Res., 4 (1989) 373.